

Furaca #06

AIM:- Preparation of Furaca from 7-ACA by new
route

MODIFICATION:-

- 1) 0.15g EDTA added before 7-ACA addition.
- 2) 0.15g EDTA added before Reaction mass addition.
- 3) 25% excess NaSH.

PROCEDURE:-

Stage I

Preparation of TFA

RAW MATERIALS:-

2-Furoyl chloride	: 27.5g	(23ml)
DMW	: (350 + 15) ml	
NaSH	: 37.5g.	
EtOAc	: 250 ml	
1:1 HCl	: 49 ml.	pH - 0.94.
DMW	: 175 ml.	
NaHCO ₃	: 20 g.	
1:1 HCl	: 44 ml.	pH - 0.95
EtOAc	: 100 ml	

PROCEDURE:-

- 1) DMW was charged followed by NaSH at RT.
- 2) The funnel was washed with DMW.
- 3) 2-Furoyl chloride was then added in 40-45' at 20-22°C.
- 4) The mixture was stirred for 5' and sample given for monitoring.
- 5) EtOAc was then added and the pH adjusted to 0.9-1.0 with 1:1 HCl in 15-20'.
- 6) The layers were separated and OL1 given for HPLC.
- 7) To the organic layer DMW was added and the pH adjusted to 7.0-7.2 using NaHCO₃ in 15-20' at 20-22°C.
- 8) The mixture stirred for 30' at 20-22°C.

8) The layers were separated. To the aqueous layer EtOAc was added the pH adjusted to 0.9-1.0 using 1:1 HCl.

9) The layers were separated and the organic layer (OL₂) taken for next stage after a sample was given for HPLC.

Reaction monitoring

	TFA	Imp.
	99.51	-
OL ₁	98.17	1.06
OL ₃	96.50	1.98

Observations

OL₁ - 250 ml AL₂ - 230 ml OL₃ - 130 ml

Stage II

Preparation of Fuzara..

RAW MATERIALS

7-ACA	:	50.0 g.
EtOAc	:	200 ml.
GAA	:	30 ml
BF ₃	:	68.5 g
TFA	:	130 ml.
DMW	:	150 ml.
EDTA	:	(0.15 + 0.15) g.
SHS	:	1.0 g
20% NH ₃	:	87 ml
DMW	:	(50 + 150 + 50) ml. (Spray + Slurry + Spray)
EtOAc	:	(50 + 150 + 50) ml. (Spray + Slurry + Spray)

PROCEDURE:-

1) EtOAc and GAA were charged and the temperature lowered to 0°C.

2) BF₃ was then purged at 15°C.

3) EDTA was then added.

4) The mixture stirred for 5'

- 5) 7-ACA was then charged followed by TFA and the mixture stored till the completion of the reaction.
- 6) DMW was cooled to 15°C separately.
- 7) After completion of the reaction, EDTA was added followed by the ^{mass} to DMW. Reaction was then charged followed by SHS.
- 8) The pH was then adjusted to 3.5 in 30-45' using 20% NH₃ at 25-35°C.
- 9) The mixture was stirred for 30' at 25-25°C. 20-25°C.
- 10) The product was filtered and washings with DMW and ~~and~~ given.

Reaction monitoring.

	7-ACA	Furaca	TFA	Imp.
45'	9.31	72.77	16.38	0.24
1 hr 45'	1.75	87.2	8.67	0.33
2 hr 15'	0.91	88.77	7.84	0.33

wet wt : 116.8 g.

This FURACA is used for CFUR # 05